YES NO N/A

CASE NUMBER:_____ LAB:_____

SITE:_____

1.0 Introduction

1.1 The attached Standard Operating Procedure (SOP) is applicable to nitro substituted aromatics and nitro substituted amines by High Performance Liquid Chromatography (HPLC) data. Its scope is not only to facilitate the data validation process of the data reported by the contracting laboratory but also to ensure that the data is being reviewed in a uniform manner.

1.2 The SOP is based upon the quality control and quality assurance requirement specified in the analytical **Method 8330, November 1992 (Revision 0).**

2.0 Responsibilities

2.1 The reviewer must be knowledgeable of the analytical method and its QC criteria.

2.2 The reviewer must complete and/or file the following:

Data Assessment Checklist- The data reviewer evaluates each criterion carefully and checks if data is in compliance, non-compliance or not applicable.

Data Assessment Narrative- The data reviewer must present professional judgement, address areas of concern and comment on the validity of the overall data package. The reviewer must explain the reasons for rejecting and/or qualifying the data.

Rejection Summary Form-The reviewer must submit a completed form using a ratio format.

Organic Regional Data Assessment Summary- A completed

YES NO N/A

form must be submitted.

Telephone Record Log-All phone conversations must be transcribed by the reviewer. Upon completion of the data review, the original telephone log is attached to the data assessment narrative.

3.0 Data Completeness and Deliverables

3.1 Have any missing deliverables been received and added to the data package? [ ] ___ ___

ACTION: Call lab for explanation/resubmittal of any missing deliverables. If lab cannot provide them, note the effect on review of the package under the "Contract Problems/Non-Compliance" section of reviewer narrative.

3.2 Was SAS-request included with package? [ ] ___ ___

If no, a SAS-request can be retrieved from RSCC.

4.0 Cover Letter SDG Narrative

4.1 Is the Narrative or Cover Letter Present? [ ] ___ ___

4.2 Are Case Number and/or SAS number contained in the Narrative or Cover letter? [ ] ___ ___

5.0 Traffic Reports and Laboratory Narrative

5.1 Are Traffic Report Forms present for all samples? [ ] ___ ___

ACTION: If no, contact lab for replacement of missing or illegible copies.

YES NO N/A

5.2 Do Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data? ___ [ ] ___

NOTE: Samples are not preserved.

ACTION: If samples were not iced upon receipt at the laboratory, flag all positive results "J" and all non-detects "UJ".

YES NO N/A

NOTE: Samples must be kept below 4°C from the time of collection through analysis. Degradation of some compounds occurs with heat. Samples should not be exposed to sunlight or temperatures above room temperature.

6.0 Special QC

6.1 Prior to preparation of stock solutions, acetonitrile, methanol, and water should be analyzed to determine possible interferences with analyte peaks. A different batch of solvent should be used if contamination is present.

6.2 Chromatograms are to be submitted showing that there are no interferences with analyte peaks.

Are these chromatograms present in package? [ ] ___ ___

Are the chromatograms free of interferences? [ ] ___ ___

ACTION: Ask lab for resubmittals. If deliverables are unavailable, judge the effect on the validity of the data. If questionable, contact SMO and note in data assessment.

7.0 Holding Times

7.1 Have any technical holding times, determined from date of collection to date of analysis, been exceeded?

A. For aqueous samples 7 days from sample collection to extraction ___ [ ] ___

B. For soil/sediment samples 14 days from sample collection to extraction ___ [ ] ___

C. For all samples extracts 40 days from time of extraction to time of analysis ___ [ ] ___

YES NO N/A

Holding Time Violations
Table 1

(See Traffic Report)

Sample ID	Date Sampled	Date Received	Date Lab Extracted	Date Analyzed

ACTION: If technical holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded. The reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results must be qualified "J", but the reviewer may determine that non-detect data are unusable (R).

8.0 Surrogate Recovery

8.1 Are the Organic Analysis Data Sheets (Form I) present and complete with surrogate recoveries

YES NO N/A

for each of the following matrices:

a. Water? [ ] ___ ___

b. Soil/Solid? [ ] ___ ___

ACTION: Call lab for explanation/resubmittals. If missing deliverables or information are unavailable document the effect in the data assessment.

YES NO N/A

8.2 Was the surrogate % recovery out of specifications for any field sample, method blank or QC sample? ___ [ ] ____

8.3 Were surrogate retention times (RT) within ± 1.0% of the mean surrogate RT calculated from the initial calibration? [ ] ___ ___

ACTION: Circle outliers in red.

ACTION: If the retention time shift (RTS) or % recovery of the surrogate in the field or QC samples is out of specification, then all associated sample data should be qualified. Use **Table 2** below as a guide.

Surrogate Recovery

TABLE 2

The following table summarizes the surrogate recovery criteria and the data qualification guidelines for all associated field samples.

SURROGATE	NOT QUALIFIED	J	R	N
% RECOVERY - FIELD SAMPLES				
Detects	50 - 125%	< 50%; > 125%		
Non-detects	$ 10%	< 10%*	< 10%*	
% RECOVERY - BLANKS AND QC SAMPLES **				
Detects	50 - 125%	< 50%; > 125%		
Non-detects	$ 20%	10 - 19%	< 10%	
RTS - field samples	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *
RTS - QC samples	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *

* **Use professional judgement.**

YES NO N/A

** **If the surrogate recovery in a QC sample or blank was less than 50 percent or greater than 125 percent, then the field sample data should be qualified if the surrogate in the field sample is outside 50 - 125 percent recovery.**

YES NO N/A

8.4 Are there any transcription/calculation errors between raw data and surrogate recoveries on Forms: [ ]

ACTION: If large errors exist, call lab for explanation/ resubmittal, make necessary corrections and note errors in the data assessment.

9.0 QC Check Reference Sample

9.1 Is the QC Check Reference Sample Recovery Form present? (Created by Lab) [ ]

9.2 Was the QC Check Reference Sample analyzed at the required frequency (once per SDG or every 20 samples, for each matrix? [ ]

NOTE: The QC Check Form has to be created by the Lab. Concentration of the spiking solution is 5-10 times the estimated quantitation limits for all analytes listed in section 12 of the SAS.

NOTE: QC Check Reference Sample information is important, data is used to judge extraction efficiency.

ACTION: If any QC Check data are missing, call the lab for explanation/resubmittal. If the lab cannot provide missing deliverables, document the effect on the validity of the data in the data assessment. Positive hits should be flagged "J" and non-detects should be flagged "R".

9.3 How many QC check recoveries are outside QC limits of 70-125%?

Water	Soil
_____ out of ___	_____ out of ___

YES NO N/A

ACTION: If any recovery is greater than 125%, positive results should be flagged "J" for the affected compound.

YES NO N/A

When two or more analytes have recoveries above 10% but below 70% all associated data should be flagged "J" (positive and non-detects).

When one or more analytes have recoveries below 10% qualify all associated data "R".

Note in data assessment. If further assistance is required, contact SMO for instructions.

It should be noted for TPO action,if a laboratory fails to analyze a QC Check Reference Sample or if a laboratory consistently fails to generate acceptable recoveries.

10.0 Calibration Data

10.1 Initial Calibration

The initial calibration standards must be analyzed daily prior to any sample analysis. The lab may have to create two sets of initial standard solutions if several analytes coelute.

The initial calibration curve must be injected in triplicate for each of the 5 levels.

10.2 Are the chromatograms and data system printouts (Quant reports) present for initial and triplicate calibrations? [ ] ___ ___

10.3 Are the modified Initial Calibration Forms (Pest VI-1, VI-2 Forms) present? [ ] ___ ___

10.4 Are modified Forms present and completed for each analytical sequence and each column? [ ] ___ ___

YES NO N/A

ACTION: If any Calibration Standard chromatograms and forms are missing, contact the lab for resubmittals.

10.5 Are there any transcription/calculation errors between raw data and Forms? ___ [ ] ___

YES NO N/A

ACTION: If large errors exist, call lab for explanation/resubmittal. Make necessary corrections and document effect in data assessment. Check at least 2-3 values from each calibration standard and more if errors are found.

10.6 Do all standard retention times, including each compound in each level, fall within the windows established during the initial calibration analytical sequence? [ ] ___ ___

NOTE: All injections are taken into account: A total of 15 injections (three injections for each of the five levels). If the lab had to run two sets of standards there would be a total of 30 injections.

ACTION: The average retention time (RT) for each analyte is determined from the mean of the triplicate injection for each 5 point level of the initial calibration. The RT window for each analyte is equal to the average RT $\pm$ three (3) times the standard deviation. If the standard deviation comes out to equal zero, the window will be $\pm$0.08. The (Pest form VI-1) Form should be modified to include the average of the three injections for each of the 5 levels.

10.7 Are the linearity criteria for the initial analyses within limits for both columns? (%RSD must be $\leq$20%) ___ [ ] ___

ACTION: If no, qualify all associated positive results generated during the sequence "J" and all non-detects "UJ". When %RSD >90%, flag all non-detects results for that analyte as unusable "R".

10.8 Do the standards show degradation products of tetryl? ___ [ ] ___

YES NO N/A

NOTE: Degradation products of tetryl appear as a shoulder on the TNT peak. Peak heights rather than peak areas should be used when tetryl is present in concentrations that are significant relative to the concentration of TNT.

ACTION: If degradation is detected or suspected, qualify positive hits for tetryl and TNT as "J". If degradation is suspected, and tetryl is reported as non-detected, qualify the analyte as "UJ". Note in data assessment.

11.0 Continuing Calibration

11.1 Are Forms (Modified Pest VII-2) present for each continuing calibration? [ ] ___ ___

11.2 Are there any transcription/calculation errors between raw data and forms? ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittals. Make any necessary corrections, and document effect in data assessment. Check at least 2-3 values from each calibration standard and more if errors are found.

11.3 Were continuing calibrations analyzed at the required frequency? (standard should bracket every 10 samples). [ ] ___ ___

ACTION: Criteria must be met. Determine effect on data. At a minimum, all data should be qualified "J", but the reviewer may determine that data are unusable "R". Document the data qualifications in data assessment.

11.4 Do all standard retention times for

YES NO N/A

each continuing calibration fall within the windows established by the initial calibration? [ ] ___ ___

ACTION: If no, beginning with the samples which followed the last in-control standard, check to see if the chromatograms contain interfering peaks. If peaks are present, qualify positive results and non-detects for analytes outside their respective windows as unusable "R".

11.5 Are the RPD values for all continuing calibration standards ≤15%? [ ] ___ ___

ACTION: If the %RPD is >15% for the analyte being quantitated, qualify all associated positive results "J" and non-detects "UJ". The "associated samples" are those which followed the last in-control standard up to the next passing standard. If the %RPD is >90%, flag all non-detects for that analyte unusable "R".

11.6 Compare the peak heights of continuing calibration checks obtained during the day with peak heights obtained from first calibration check of the sequence.

Is the percent RPD for peak heights less than 20% for each of the analytes of each of the Continuing Calibration Checks? [ ] ___ ___

ACTION: Use professional judgement to verify the reliability of the data.

12.0 Analytical Sequence

12.1 Is Form VIII present and completed for each column and each period of analyses? [ ] ___ ___

ACTION: If no, contact lab for resubmittals.

12.2 Was the proper analytical sequence followed

YES NO N/A

for each initial calibration and subsequent analyses? [ ] ___ ___

NOTE: Sequence is as follows: 5 point Initial Calibration, Method Blank, Laboratory Control Sample (LCS), Continuing Calibration, 10 sample extracts, Continuing Calibration, 10 sample extracts and so on. The sequence must always end with a Continuing Calibration. All sample extracts, Method Blanks, LCS, and continuing calibrations must be analyzed within 24 hours after the analysis of the Initial Calibration Standards and the calculation of the RT windows.

ACTION: Flag "J" all data generated outside an acceptable twenty-four hour sequence starting after the calculation of the RT windows resulting from the initial calibration unless the daily calibration meets the calibration criteria for all target analytes. If the daily calibration standard does not meet the criteria for some analytes the reviewer might reject

those values. In any case, if the 24 hour sequence is exceeded it should be noted in the data assessment under contract non-compliance.

13.0 Method Blanks

13.1 Is the Method Blank Summary (Form IV) present for each of the following matrices?

a. water [ ] ___ ___

b. soil [ ] ___ ___

13.2 Frequency of Analysis:

Has a Method/Prep blank been analyzed at the same time for each batch of samples extracted. [ ] ___ ___

YES NO N/A

ACTION: If any method blank data are missing, call lab for explanation/resubmittal. If method blank data are not available, reject "R" all associated positive data.

13.3 Are the positive hits present in the blank less than detection limit? [ ] ___ ___

ACTION: If hits are ≥ detection limits, analysis should be discontinued. All associated samples must be re-prepared and re-analyzed with a new blank. If lab did not follow this action, note in data assessment. Follow guidelines under contamination section.

13.4 Chromatography: review the method blank raw data chromatograms, quant reports or data system printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable? [ ] ___ ___

ACTION: Use professional judgement to determine the effect on the data.

YES NO N/A

14.0 Contamination

NOTE:"Water blanks", "drill blanks", and distilled water blanks" are validated like any other sample, and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.

14.1 Did the lab run an instrument blank following a sample analysis which contained an analyte(s) at high concentration(s)to evaluate possible sample cross-contamination? [ ] ___ ___

ACTION: Sample analysis results after the high concentration sample must be evaluated for carryover. Sample cross-contamination should be noted for TPO action if an effect on the data is suspected. An Instrument Blank is not required in the methodology.

14.2 Do any Method Blanks have positive results (TCL)? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor. ___ [ ] ___

14.3 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: Note in data assessment that there is no associated field/rinse/equipment blank.

14.4 Do any field/rinse blanks have positive results (TCL)? ___ [ ] ___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated with a

YES NO N/A

particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate recovery, instrument performance criteria, or calibration QC problems.

YES NO N/A

ACTION: Follow the directions in **Table 3** below to qualify TCL results due to contamination. Use the largest value from all the associated blanks. If any blanks are grossly contaminated, all associated data should be qualified as unusable (R) (see item # 12.3).

Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL & > 5x blank value
Flag sample result with a "U"	Report CRQL & qualify "U"	No qualification is needed

NOTE: Analytes qualified "U" for blank contamination are still considered as "hits" when qualifying for calibration or other QC criteria.

15.0 Target Compound List (TCL) Analytes (Form I)

15.1 Are the Organic Analysis Data Sheets (Form I) present with required header information on each page, for each of the following:

a. Samples ? [ ] ___ ___

b. Laboratory Control Samples? [ ] ___ ___

c. Blanks? [ ] ___ ___

d. Matrix spikes and matrix spike duplicates? [ ] ___ ___

e. Lab duplicate? [ ] ___ ___

YES NO N/A

YES NO N/A

15.2 Are the Chromatograms, and the data system printouts (Quant Reports) included in the sample package for each of the following:

a. Samples? [ ] ___ ___

b. Laboratory Control Samples? [ ] ___ ___

c. Blanks? [ ] ___ ___

d. Matrix spikes and Matrix spike duplicates? [ ] ___ ___

e. Lab duplicate? [ ] ___ ___

ACTION: If any data are missing, Contact Lab for resubmittals.

15.3 Are the response factors shown in the Quant Report? [ ] ___ ___

15.4 Is chromatographic performance acceptable with respect to:

Baseline stability? [ ] ___ ___

Resolution? [ ] ___ ___

Peak shape? [ ] ___ ___

Full-scale graph (attenuation)? [ ] ___ ___

Other: _____ [ ] ___ ___

ACTION: Use professional judgement to determine the acceptability of the data.

YES NO N/A

16.0 Compound Identification

16.1 Is Form X completed for every sample in which an analyte was detected? [ ] ___ ___

ACTION: If no, contact lab for resubmittals.

YES NO N/A

16.2 Are there any transcription/calculation errors between raw data and Forms? ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittals. Make any necessary corrections and note effect in data assessment. Check at least 2-3 values from each calibration standard and more if errors are found.

16.3 Are retention times (RT) of sample analytes within the established RT windows for both analyses? [ ] ___ ___

ACTION: Qualify as unusable "R" all positive results which were not confirmed by second HPLC column analysis. Also qualify as unusable "R" all positive results not meeting RT window unless associated standard compounds are similarly biased. The reviewer should use professional judgement to assign an appropriate quantitation limit.

16.4 Is the percent difference (%D) calculated for the positive sample results on the two HPLC columns <25%? [ ] ___ ___

ACTION: If the reviewer finds neither column shows interference for the positive hits, the data should be flagged as follows:

% Difference	Qualifier
25-90%	J
>90%	R

NOTE: The lower of the two values is reported on Form I. If using professional judgement, the reviewer determines that the higher

YES NO N/A

result was more acceptable, the reviewer should replace the value and indicate the reason for the change in the data assessment.

YES NO N/A

16.5 Check chromatograms for false negatives, interferences and degradation. Tetryl decomposes rapidly in methanol/water solutions as well as with heat. All samples expected to contain tetryl should not be exposed to temperatures above room temperature. Degradation products of tetryl appear as a shoulder on the TNT peak. Peak heights rather than peak areas should be used when tetryl is present in concentrations that are significant relative to the concentration of TNT.

ACTION: Use professional judgement to determine qualification of analytes.

17.0 Compound Quantitation and Reported Detection Limits

17.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the calculations were adjusted for percent moisture. Were any errors found? ___ [ ] ___

NOTE: Single peak analyte results can be checked for rough agreement between quantitative results obtained on the two HPLC columns. The reviewer should use professional judgement to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interfering compound is indicated, the lower of the two values should be reported and qualified as presumptively present at an approximated quantity "JN". This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate that

YES NO N/A

the presence of interferences has interfered with the evaluation of the second column confirmation.

17.2 Are the CRQLs adjusted to reflect sample dilutions for each Form I? [ ] ___ ___

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note in data assessment.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and its associated value on the original Form I and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red "X" with a red pencil across the entire page of all Form I's that should not be used, including any in the summary package.

18.0 Lab Duplicates

18.1 Were any lab duplicates submitted for for each extraction batch?
a. waters [ ] ___ ___
b. soils [ ] ___ ___

18.2 Are the Relative Percent Difference (RPD) values for the analytes reported in the duplicate samples <20%? (depending on the SAS-request; the criteria might be <40%). [ ] ___ ___

ACTION: Compare the reported results for lab duplicates and calculate the relative percent difference.

YES NO N/A

ACTION: Any gross variation between duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of lab duplicates should be confirmed by contacting the lab.

ACTION: Flag all associated data with an * for "out of control" duplicate.

19.0 Matrix Spikes

19.1 Is the Matrix Spike/Matrix Spike Duplicate recovery Form present? [ ] ___ ___

YES NO N/A

19.2 Were the matrix spikes analyzed at the required frequency? (one per matrix and per SDG).] ___ ___

ACTION: If any matrix spike data are missing, contact the lab for resubmittals.

19.3 The matrix spike consists of all target analytes at concentrations 5-10 times the estimated quantitation limits listed in Table 1/Method 8330.
Were the correct analytes and were the concentrations within range? [ ] ___ ___

NOTE: Field blanks cannot be used for Matrix spike analysis. If field/rinse blanks were MS/MSD, then make note under Contract non-compliance.

19.4 How many spike recoveries are outside the QC limits of 50-140%?

Water	Soil
_____ out of ___	_____ out of ___

19.5 How many RPD's for Matrix Spike and Matrix Spike duplicate recoveries are outside QC limits of 20% (depending what is stated in the SAS-request)?

Water	Soil
_____ out of ___	_____ out of ___

ACTION: Flag all associated data "N" for "out of control" matrix spike for the field sample used for the MS/MSD.

ACTION: No additional action is taken on MS/MSD data alone. However using informed professional judgement, the data reviewer

YES NO N/A

may use the matrix spike data results in conjunction with other QC criteria and determine the need for some qualification of the data.